Analytical Methods

ROYAL SOCIETY OF CHEMISTRY

TECHNICAL NOTE

View Article Online
View Journal | View Issue

Cite this: *Anal. Methods*, 2022, **14**, 581

Improving particle collection efficiency of sampling wipes used for trace chemical detection†

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Received 21st September 2021
Accepted 14th December 2021

DOI: 10.1039/d1ay01609a

rsc.li/methods

Improvement of the particle collection efficiency of sampling wipes is desirable for optimizing the performance of many wipe-based chemical analysis techniques used for trace chemical screening applications. In this note, commercially available Teflon coated fiberglass and calendered Nomex sampling wipes were modified by mechanically scoring the wipe surface to produce topography that promoted enhanced and localized particle collection. Wipe surface modifications improved particle collection efficiency, relative to unmodified wipes, by factors of 3 to 13 depending on sampling conditions, wipe type, and surface sampled. Improvements were demonstrated for both model polystyrene latex microspheres and inkjet printed explosive particles. The modifications also concentrated particles into pre-defined locations on the wipe which can be engineered to ensure maximum overlap with the thermal desorber of a trace contraband detection system allowing for more effective analysis of collected trace residues.

Background

Screening of people and their belongings (*i.e.*, luggage, vehicles, and packages) for trace explosive and narcotic residues (trace contraband) is commonplace in aviation security and law enforcement environments. In typical scenarios, trace contraband, in the form of non-visible, micrometer-sized particles, is collected from suspect surfaces using a dry wipe, which is directly inserted into a thermal desorber (heating element) located in the inlet of a detection system. The thermal desorber converts solid particles into vapors that are aerodynamically transported into the detector for chemical analysis using ion mobility spectrometry (IMS), mass spectrometry (MS) or amplified fluorescent polymer (AFP) based chemical detectors. Positive identification of chemical signatures from a targeted compound on a person or their belonging suggests potential contact with bulk levels of contraband material and may prompt additional screening procedures or denial of entry into a facility or venue.

While many different materials can be used for sampling wipes, some practical considerations for their selection and design include efficient particle collection, low cost, robustness and reusability, thermal stability at high temperatures, and low chemical background when heated. A woven fabric made from Teflon-coated fiberglass (TCF) is a common wipe material for both IMS and MS based detectors. TCF provides many of the desirable characteristics listed above including temperature resistance, minimal chemical background, and reusability. Another commonly used wipe material appropriate for trace detection is a meta-aramid polymer with high heat resistance (Nomex). In sheet form, it is used primarily with AFP trace detection systems. The sheets are prepared by a calendering process, where material is passed through high-pressure rollers, resulting in a flat, smooth calendered Nomex (CN) wipe material.

Efficient wipe sampling for screening applications presents two primary challenges. The first is the limited sampling time, typically a few seconds, imposed by the requirement to maintain a high throughput of people and property through screening checkpoints. Successful identification of potential threats may be impacted by the limited surface area on a suspect person or belonging that can be interrogated in the available time. Given this constraint, sampling wipes with the highest possible particle collection efficiency are beneficial for increasing the probability of a positive detection during screening. Previous studies,[1] and the results discussed herein, demonstrate that particle collection efficiencies (PCE) of the CN and TCF materials are often 10% or less, indicating that further optimization of PCE would be desirable. The second challenge is that, even when contraband particles are collected on the sampling wipe, they may not be desorbed, and therefore detected, because the collection area of the wipe is often larger than, or may be misaligned with, the footprint of the thermal desorber within the trace detection system.[2] This mismatch is

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† Electronic supplementary information (ESI) available. See DOI: 10.1039/d1ay01609a

due to requirements to physically hold the wipe, either with a mechanical wand or by hand, thus restricting the available collection area to a portion of the wipe. Additional detector-related restrictions regarding power consumption and aerodynamic collection volumes may limit the overall size of the desorber and, subsequently, the area of the wipe that can be desorbed and analyzed. The discrepancy between the area of the wipe containing collected particles and the area interrogated by the trace detector can lead to incomplete analysis of the collected sample, possibly resulting in decreased probability of detection.[2]

The combined challenges of limited analysis time, low particle collection efficiencies and the potential for incomplete sample analysis provide the motivation for exploring wipe modifications for trace contraband sampling. Potential strategies for such modifications are informed by recent observations of the wipe sampling process using *in situ* optical microscopy[3] and high-speed microscopic imaging to observe particle–wipe interactions during particle collection by swiping.[4] Both studies highlight the important role of a wipe's surface topographical features in promoting particle collection. Due to the weave of the underlying fiberglass of the TCF wipe, it's surface can be characterized as a series of peaks and valleys where the peaks (typically 60 μm to 90 μm higher than the valleys) act as the primary contact points with the sampled surface. It has been observed that both contraband particles and PSL microspheres can be channeled between the surface contact points and through the valleys of the wipes without being efficiently collected.[4] In the case of CN wipes, a lack of significant macroscopic surface topography may allow particles to translate across the surface of the wipe without being retained on an appropriate portion of the wipe surface.[4]

A common strategy for improving particle collection efficiency of sampling wipes is to use solvent wetted wipes which can provide particle collection efficiencies greater than 90% on non-porous surfaces.[5] Wetted wipes are used in several non-screening applications of trace detection including analysis of hazardous drug levels in medical facilities[5] and measurement of residual methamphetamine contamination in clandestine drug laboratories.[6] Unfortunately, this approach is not compatible with current trace screening applications which require dry wipes due to the deleterious effect of solvents on ionization chemistry used in widely deployed IMS detectors.

Previous studies have explored several methods for improving the collection efficiency of dry wipes including chemical modification of the wipe surface,[7] addition of surface topography through abrasion[8–10] or addition of an adhesive layer to promote particle retention on the wipe.[11] These methods each have merit but were not specifically designed to focus particle collection onto targeted regions of the wipe. These methods may also add significant cost and complexity to manufacturing along with the potential of introducing chemical background into the trace detector. In this technical note, we describe a simple approach for increasing PCE on specific regions of a dry wipe by using a commercial razor cutter to score the surface of the wipe in defined patterns. This approach is easy to implement, cost effective, and requires no chemical modification that might affect performance by IMS, MS or AFP detection. The utility of the approach is demonstrated for TCF and CN wipes but may be generally applicable to other dry wipe materials. The potential mechanism leading to improvement is described, and measurements of PCE using PSL microspheres and inkjet printed explosive particles are given.

Experimental methods

Wipe and test surface materials

Teflon-coated fiberglass (TCF) wipes were purchased from DSA Detection (North Andover, MA) and calendered Nomex (CN) wipes from FLIR systems (Nashua, NH).‡ Test surfaces for PCE experiments included acrylonitrile butadiene styrene (ABS) plastic (TAP Plastics, Seattle, WA), synthetic leather–vinyl (referred to hereafter as vinyl) (fabric.com), ballistics nylon (Seattle Fabrics, Seattle, WA), and 304 stainless steel with a brushed (number 4) surface (Stainless Supply, Monroe, NC). All test surfaces were cleaned with ethanol (ACS reagent >99.5%, Sigma-Aldrich, St. Louis, MO) and cut into 5 cm by 15 cm sized strips prior to use.

Preparation of wipe materials

A Silhouette Cameo desktop blade cutter (Silhouette America, Lindon, UT) was used for scoring the TCF and CN scored wipes. Settings for the blade cutter included (arbitrary units): blade 10, speed 5, force 20 and 2 passes/pattern. In preliminary studies (not reported here) we used microscopy to examine particle collection of PSL's on 12 different patterns cut into the fiberglass wipes. These patterns included holes, diagonal cuts and lines both parallel and perpendicular to the swiping direction with varying number of lines. While not intended to be a quantitative study due to a limited number of replicate measurements, we did make the general observation that lines cut perpendicular to the direction of swiping (perpendicular to the long axis of the swipe) appeared to provide higher PCE with a trend of greater PCE with increased number of lines. We selected 5 lines for our experiments as providing the greatest PCE without compromising the robustness of the wipe as it was found to be prone to tearing during use with higher numbers of lines. Examination of the 3D topography of the scored wipes was conducted using a Zeiss Smartzoom 5 automated digital inspection microscope (Carl Zeiss Microscopy, Thornwood, NY).

Particle collection efficiency measurements using polystyrene latex spheres

Monodisperse Fluoresbrite polystyrene latex (PSL) microspheres (Polysciences, Inc, Warrington, PA 20 μm in diameter) were prepared from a water suspension by filtering through

‡ Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by NIST, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

a 2.54 cm-diameter Nuclepore track-etch membrane filter (Corning Inc, Corning, NY) after which they were allowed to dry. PSL microspheres were then transferred by lightly touching the filter onto the test surface. The primary test surface used for the PSL PCE measurements using TCF wipes was vinyl. Test surfaces were secured to a flat stainless steel substrate using double stick tape to aid in bringing the entire surface into a single focal plane under the microscope. An infinity 1-2CB, two mega pixel camera (Teledyne Lumenera, Ottawa, Ontario CA) mounted on a Zeiss SteREO Discovery V8 fluorescence microscope (Carl Zeiss Microscopy, Thornwood, NY) was used for imaging and was equipped with an automated Prior ProScan X-Y-Z sample stage (Prior Scientific Inc., Rockland, MA) to allow for image tiling. Image tiling allowed for the creation of a mosaic of contiguous high-magnification microscope images covering the entire microsphere deposit area. Image capture, stage control, and particle identification was accomplished using the Image Pro software package (Media Cybernetics, Rockville, MD). Microsphere localization and counting utilized the automated bright object image threshold setting in Image Pro with exposure time, gain, gamma and offset adjusted for the best quality images.

Once the initial PSL microsphere deposit was imaged and individual microspheres were counted, the vinyl test surface was swiped using a slip/peel tester (IMASS Inc., Accord, MA) using experimental conditions described previously.[12] A mass of 660 g was used to apply a load on the wipe surface of 6.5 N, using a translational velocity set to 5 cm s^{-1} over a 12 cm length of the test surface. After completion of the swiping experiment, the test surface was re-imaged to obtain the PSL particle collection efficiency PSL-PCE according to eqn (1).

$$\text{PSL particle collection efficiency (\%)} = \frac{\text{Microsphere count on wipe}}{\text{Microsphere count on test surface}} \times 100 \quad (1)$$

Multiple trials ($n = 5$ to 18) were conducted for each experiment with the mean and standard deviation presented. Because the coordinates of each counted microsphere were measured, the number of microspheres collected in specified regions of interest on a wipe were also determined.

Particle collection efficiency from quantitative analysis of collected explosive particles

A second method for determination of the PCE of the different wipes involved using inkjet-printed explosives particles and quantitative analysis by extraction and electrospray ionization mass spectrometry (ESI-MS). Experimental details for these procedures may be found in several recent publications.[12–14] Briefly, explosive particle deposits were inkjet printed as 8 × 8 spot liquid droplet arrays, with a total mass loading of 350 ng, onto 1 cm by 1 cm square Bytac strips (Teflon film with foil backing, BytacBench and Shelf Protector Sheets, SPI Supplies and Structure Probe, West Chester, PA) using a JetLab 4XL inkjet printer (MicroFAB, Plano, TX) and a 1 mg mL^{-1} acetonitrile standard solution of 1,3,5-trinitroperhydro-1,3,5-triazine (RDX) (Cerilliant Corp., Round Rock, TX). After printing, the arrays were dried to create solid explosive particle residues which were then transferred to the test surface using a dry transfer protocol.[12] A subset of the Bytac strips containing explosive particles were not transferred, in order to validate the initial mass of inkjet printed RDX. A robotic wipe-sampling instrument, based on a repurposed Robo3D fused deposition modelling (FDM) printer (Robo3D, San Diego, CA), was used to complete the swiping experiments as a function of the mass placed on the mount holding the sampling wipe with values ranging from 260 g to 1060 g (applied load of 2.6 N to 10.4 N) using a translational velocity of 5 cm s^{-1} over a total travel distance of 12 cm.[14] Both this and the Bytac strip used for manual deposition of the explosive particles onto the test surface and the wipe (TCF or CN) were set aside for quantitation. Previous results have demonstrated that the slip/peel tester described previously, and the robot sampling system (which uses the same mounting hardware) provide equivalent results.[14]

Quantitation of the RDX on the Bytac strips and wipes was completed by rinsing (Bytac strips) or submerging (wipes) in 1 mL of methanol (Chromasolv grade, Millipore Sigma, St. Louis, MO) containing 25 ppb of an isotopically labeled internal RDX standard (99% $^{13}C_3{}^{15}N_3$) (Cambridge Isotope Laboratories, Andover, MA). The extracts were then transferred to 2 mL amber vials for analysis by ESI-MS using a Thermo Scientific UltiMate 3000 LC system (Thermo Scientific, Waltham, MA) coupled to a JEOL JMS-T100LP time-of-flight mass spectrometer (JEOL USA, Peabody, MA) using experimental conditions described elsewhere.[14]

RDX particle collection efficiency (RDX-PCE) was calculated as the percentage of RDX on the wipe relative to the mass of RDX deposited on the test surface (eqn (2)). The mass of RDX deposited on the test surface was the difference between the mass originally present on the Bytac strip (measured from a subset that was not used for the swiping experiment) and the amount that remained on the Bytac strip after dry transfer to the test surface. The measured extraction efficiencies from the unmodified CN wipes and the Bytac strips were greater than 98% and were not included in the calculation. The measured extraction efficiencies from the scored CN wipes ranged from 75% to 90%, depending on the pattern of scoring, and were included in the calculation.

$$\text{RDX PCE (\%)} = \frac{\text{Mass RDX on wipe}}{(\text{Mass of RDX Printed on Bytac} - \text{Mass of RDX Remaining on Bytac})} \times 100 \quad (2)$$

Results and discussion

Feasibility experiments with Teflon coated fiberglass wipes and vinyl test surfaces

Scoring of a wipe surface was hypothesized to provide improved PCE by providing asperities on the surface that act as local microparticle collectors. This is demonstrated in Fig. 1 where PSL microspheres were deposited on a vinyl test surface and swiped using the slip/peel tester using both the unmodified TCF wipe and a 5 line scored TCF wipe. For each experiment, images were obtained of the as-deposited test surface, the test surface after swiping, and the collection wipe, respectively. For this experiment, the PSL-PCEs for multiple runs were calculated as $5.1 \pm 3.6\%$ ($n = 15$) for the unmodified wipes and $41.3 \pm 10.1\%$ ($n = 15$) for the scored wipes, leading to a factor of 8 improvement in collection efficiency. The mass used on the slip/peel tester was 660 g, providing a load of 6.5 N which is within the normal range of force used during wipe sampling by untrained volunteers asked to swipe with firm pressure.[1] Fig. 2A–C show individual particle locations for the unmodified wipe and Fig. 2D–F for the 5 line score pattern before and after swiping using the slip/peel tester. The location of microspheres on the scored wipe showed a clear and enhanced collection of PSL microspheres on the scores compared to the random location of PSLs on the unmodified wipe. It was also clear that the unmodified wipe had very little interaction with the microspheres, leaving the original deposit largely intact, whereas the scored wipe moved many of the microspheres across the entire swiping distance in addition to collecting a large number.

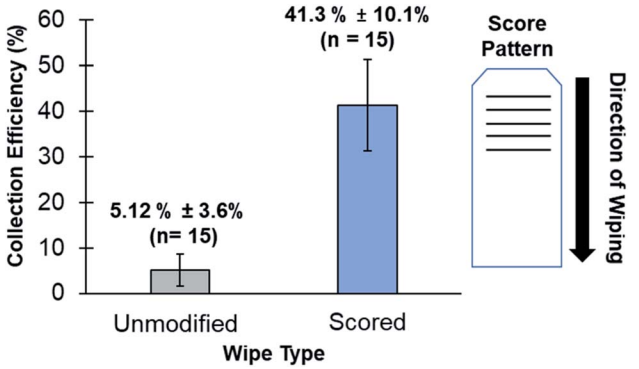

Fig. 1 Comparison of PSL-PCE from unmodified (control) and scored wipes calculated using particle counting microscopy. Uncertainties represent one standard deviation of the mean for n analyses. A schematic of the scored wipe is shown to the right of the plot with scores indicated by the black lines.

Experiments with calendered Nomex wipes

Experiments with TCF wipes demonstrated the feasibility of the score wipe approach to improve PSL-PCE when sampling a vinyl test surface. Studies were also completed to determine if the increase in PCE realized by the scored TCF wipes would translate to CN wipes. The benefits of the score line modification were qualitatively confirmed as shown in Fig. 3 which compares images of fluorescent PSL microspheres collected from a vinyl surface on an unmodified CN wipe as well as a 5 score line modified CN wipe using manual wipe sampling (with the score lines perpendicular to the direction of travel). For these wipes, the score pattern was adapted to account for the shape and location of the thermal desorber in the AFP detector. Similar to what was observed with TCF wipes, scoring produces

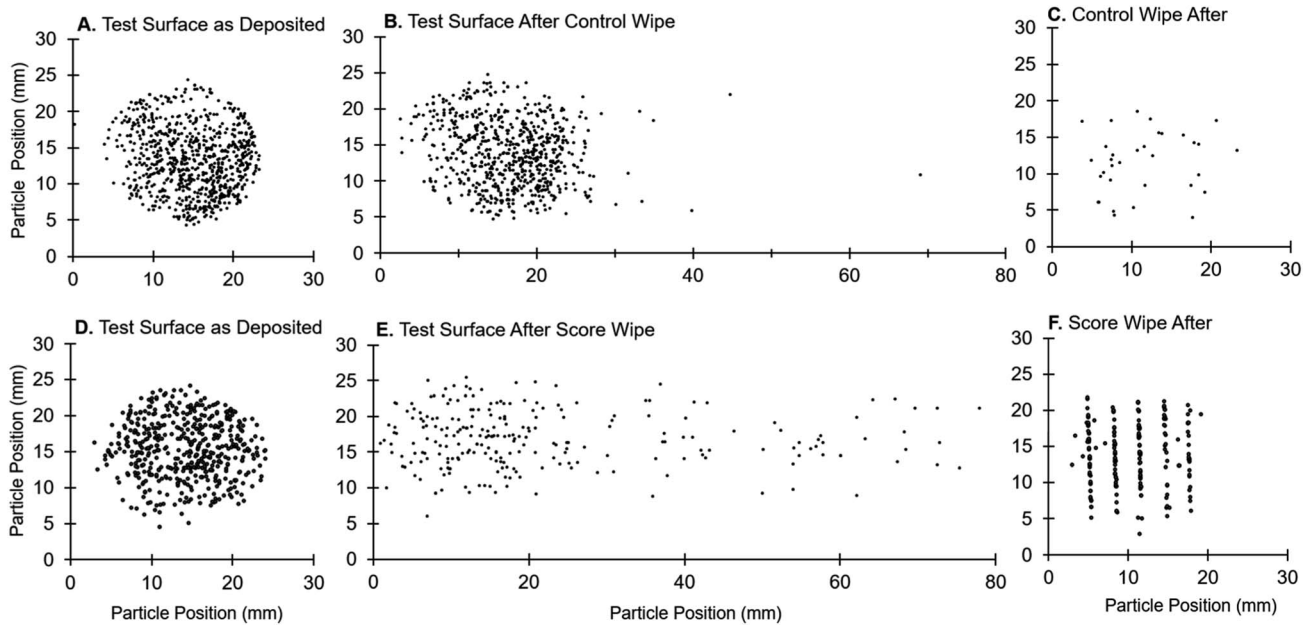

Fig. 2 Comparison of the spatial distribution of PSL microspheres on the vinyl test surface before (A and D) and after (B and E) wiping with both unmodified (top, A–C) and 5 line scored (bottom, D–F) TCF wipe using the slip/peel tester with 660 g weight (corresponding to a 6.5 N load). PSL distributions on the unmodified (C) and scored (F) wipes are also shown.

Fig. 3 Fluorescent optical image of two CN wipes after manual wiping a vinyl surface containing 20 μm PLS microspheres. The unmodified wipe (left) was first manually swiped over the surface of a vinyl sample containing dry deposited fluorescent PSLs. The score wipe (right) was then translated over the same location on the microsphere-containing vinyl surface without the addition of any additional PSL microspheres. The scores were produced with the razor cutting system to coincide with the limited circular area of the wipe interrogated by the thermal desorber of a typical APF system. The width of the CN wipe is 2.54 cm.

macroscopic topography on the CN wipe surface, improving the collection efficiency and concentrating the deposit in the actively heated area of the wipe, which is quite small in this instrument. As shown in ESI Fig. 1,† the cutting of scores under the conditions used in this work produced a score line with a maximum depth in the range of ~70–100 μm and a width of ~30–50 μm. Surrounding both sides of the score was a raised rim of fibrous debris which, though variable in height across the length of the score, typically protruded at least ~20–100 μm above the plane of the wipe surface. Microscopic examination suggests that while some particles are collected in the score, many particles are collected against these ridges and the protruding ridges may be the dominant region of particle collection leading to enhanced PCE.

The PCE of CN wipes has not previously been studied so we report several experiments used to systematically evaluate the PCE over a range of substrate types and applied sampling loads. For the majority of these studies, RDX-PCE measured using quantitative ESI-MS analysis was used rather than PSL-PCE to allow for greater applicability to the intended use.

Fig. 4A shows RDX-PCE data for explosive particles collected from a vinyl test surface using both scored and unmodified CN wipes as a function of the mass placed on the mount holding the sample wipe being tested. Individual masses from 260 g to 1060 g were used, providing a load increasing from 2.6 N to 10.4 N. Mass applied to the wipe holder rather than load in newtons is presented in the plots to provide a more intuitive value to the reader. We observed a general trend of increasing RDX-PCE with increasing applied mass for both unmodified and scored CN wipes. The RDX-PCE from this surface using the unmodified wipe, averaged over the range of applied masses, was approximately 5%, which is consistent with the TCF swipes discussed above. The improvement in RDX-PCE for the scored CN wipes, computed as a ratio of scored/unscored RDX-PCE, ranged from approximately 3 to 5 with a modest trend towards increased improvement at the lower masses. Fig. 4B shows results of the same study-this time using ABS plastic as the test surface, again showing a trend for increasing RDX-PCE as a function of applied mass. The scored wipes showed significant improvement in RDX-PCE over the unmodified wipes, with the highest improvement ratios of approximately 7 and 5 at the lower applied masses of 260 g and 660 g, respectively. Fig. 4C shows similar trends when ballistics nylon was used as the test surface. Finally, Fig. 4D shows results when stainless steel was used as the test surface, which also exhibited an increase in RDX-PCE with the largest improvements (a factor of 13) at the lowest applied mass of 260 g.

In all the cases studied, the scored CN wipes provided increased collection efficiencies for RDX compared to unmodified wipes, with the biggest gains in RDX-PCE occurring at lower applied masses. This can be explained by the rigid nature of CN wipes and the difficulty in making extended contact with the surface when smaller loads are applied, thus making the asperities produced by scoring even more critical to particle collection. Interestingly, studies from our laboratory found that the average load applied by practicing security screeners in the field during wipe sampling was between 400 g and 600 g (unpublished data), suggesting the improved PCE of the scored wipes at the lower applied loads may be most relevant for practical wipe sampling in the field.

Orientation effects with CN wipes

Through observation of the use of CN scored wipes in simulated operational settings, users did not always align the wipe perpendicular to the direction of the scores (along the long axis of the wipe) and in some scenarios would swipe in a direction parallel to the score lines. Swiping in this manner may decrease the benefit of the score, and therefore additional experiments were conducted to evaluate the effect of the orientation of the score pattern on PSL-PCE. Measurements made using the slip/peel tester and the vinyl surface with CN wipes scored both perpendicular and parallel to the direction of swiping are shown in Fig. 5 and compared with the unmodified wipes. As expected, the highest PSL-PCE was measured for the perpendicular score pattern (factor of 9 improvement), although there was still a modest improvement for the parallel score pattern (factor of 3) relative to the unmodified wipe. To account for possible variability in wipe orientation during swiping, prototype score patterns consisting of five concentric circles of increasing diameter or a cross hatched pattern were fabricated.

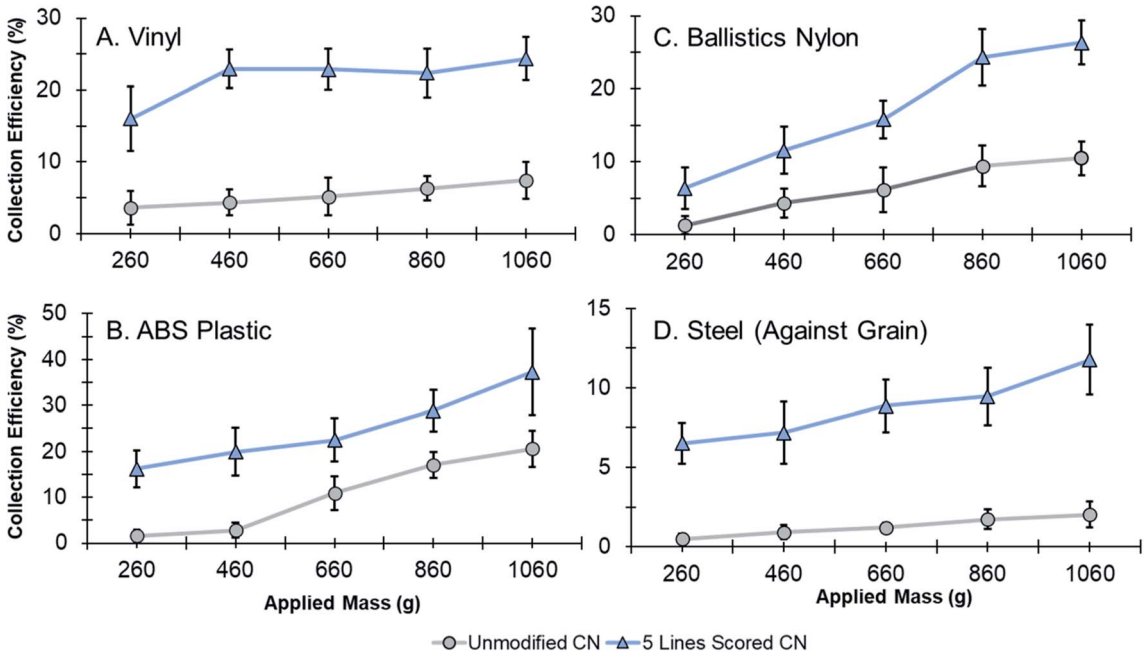

Fig. 4 RDX-PCE from vinyl (A), ABS plastic (B), ballistics nylon (C), and steel wiping against the grain (D) test surfaces comparing unmodified (grey circles) and scored (blue triangles) using CN wipes as a function of increasing mass applied to sampling wipe. Uncertainties represent one standard deviation of the mean for $n = 5$ analyses for each data point.

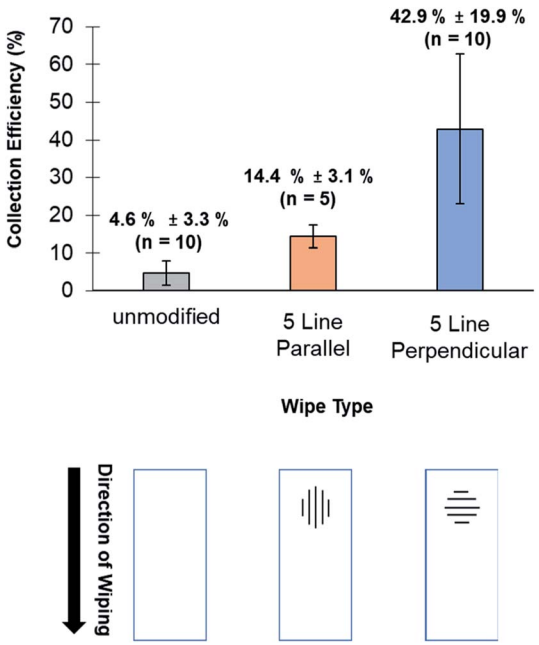

Fig. 5 Comparison of PSL-PCE for CN wipes from a vinyl test surface using unmodified wipes (grey) as well as wipes cut with 5 scores parallel (orange) and perpendicular (blue) to the direction of wiping. Experiments completed using the slip/peel tester with a 660 g applied mass (6.5 N applied load). Uncertainties represent one standard deviation of the mean for *n* analyses. Schematics of the wipes are given under their respective data points in the plot. Black lines indicate the score lines. A circular collection zone was used here to mimic the thermally desorbed area in a common APF detection system.

As shown in Fig. 6, a series of RDX-PCE measurements for explosive particles were conducted on CN wipes comparing the standard 5 perpendicular line pattern with both the cross

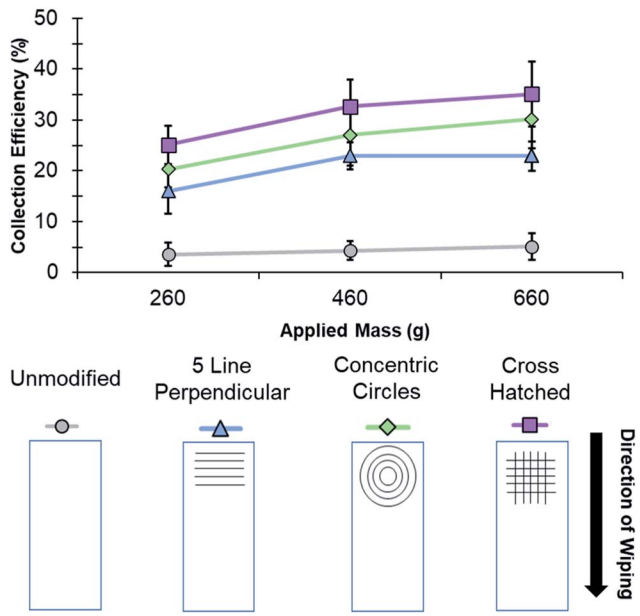

Fig. 6 PCEs for RDX from vinyl using the unmodified wipe (grey circles) and the different score patterns (5 lines (blue triangles), concentric circle (green diamonds), and cross hatched (purple squares)) on CN wipes as function of increased applied mass across applied loads from 2.6 N to 6.0 N. Schematics of the wipes used for the various experiments are shown below the plot. Black lines indicate the scores on the wipe.

hatched and concentric circle patterns with swiping along the long direction of the wipe. Both new patterns provided comparable, or modest improvements in RDX-PCE compared to the 5 line pattern. This slight increase in PCE for these new patterns compared to the simple 5 line score likely results from the increased scored surface area produced with the more complexed pattern. As observed above, even scores oriented parallel to the swiping direction provide some increase in PCE. The modified score patterns offer the potential flexibility for orientation-independent swiping; although this will be explored in a future study.

Conclusions

Modification of both CN and TCF wipes through the addition of razor-cut score patterns was found to be a simple approach for increasing PCEs for both PSL microspheres and explosive particles on different test surfaces representative of those commonly encountered in contraband screening wipe sampling environments. In addition to providing overall improvements in PCE, the score patterns can be used to focus particle collection onto specific regions of the wipe to promote targeted collection in the region that will be actively heated by the thermal desorber of commonly used trace detection instruments, potentially providing greater probabilities for targeted compound identification. Scored TCF wipes on vinyl provided collection efficiency improvements of factors of greater than 8 for swiping of PSLs. Considering four different test surfaces over a range of five applied loads, explosives particle collection using scored CN wipes provided global mean average improvements in CE ranging from a factor of 3 to a factor of 8, with individual values being generally higher at lower applied loads. Within the constraints of the study, there were no cases where the scored wipes did not improve the PCE compared to unmodified wipes. While score wipes were specifically developed to support IMS, MS and AFP based trace detection systems, the concept described in this study should be generalizable to any other wipe based trace analysis technique.

A correlation between PCE improvement and the orientation of the scores on the swipe was found, indicating the direction of swiping is critical to realizing the full improvement in PCE. To address these issues, two dimensional patterns (*i.e.*, concentric circles or a cross hatch) may allow for the wipe to be used in any orientation. However, this has not been extensively studied and is the focus of ongoing studies.

There are several potential limitations to this work, one of which is that the influence of the scored wipes and increased PCEs on the analytical performance of common detection systems was not explored. Also, because increased particle collection efficiency is non specific, it is possible that increased collection of non-specific chemical and environmental background could result in deleterious effects for targeted compounds identification, such as increased false positive alarm rates or long instrument clear downs.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

The U.S. Department of Homeland Security Science and Technology Directorate sponsored a portion of the production of this material under Interagency Agreement IAA FETN-18-00014 with the National Institute of Standards and Technology.

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